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A Method for Testing Moisture in Dried Prunes

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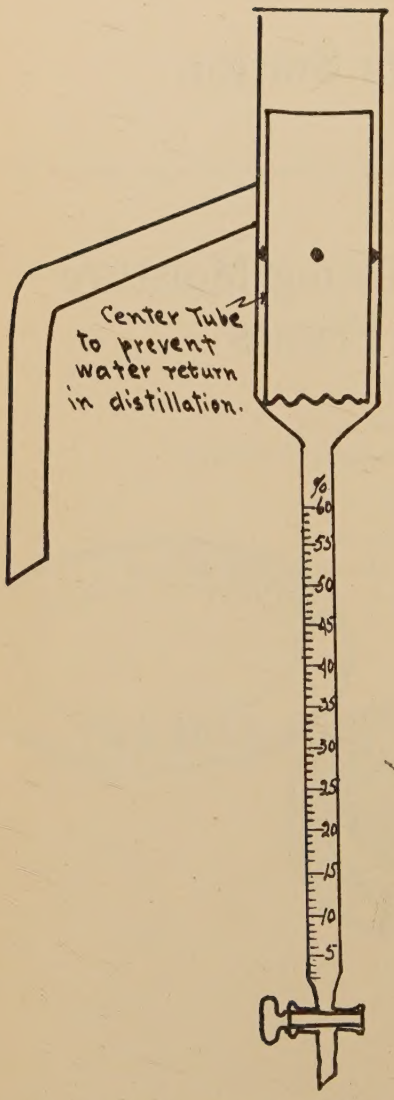

Fig. 1. Moisture tube and cylinder.

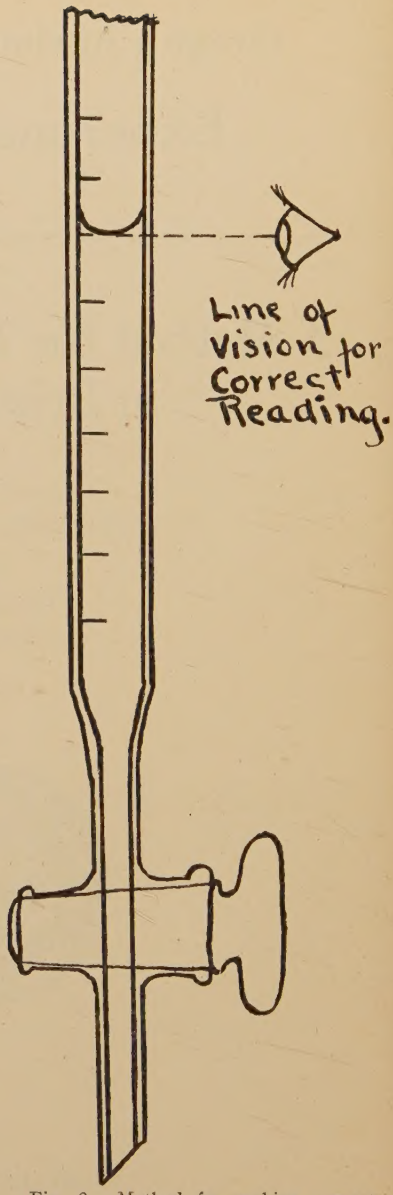

Fig. 2. Method for making correct moisture reading.

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Ernest H. Wiegand and D. E. Bullis

Lack of uniformity in the moisture content of prunes received at the packing plant is responsible for many of the difficulties occurring in storing and packing the fruit. The ordinary physical means of accurately testing fruit for moisture are entirely inadequate. Realizing that there was a need for a quicker and simpler method for the determination of moisture for both the packer and the drierman, this Station tested out the method used for determination of moisture in other products. These were not found applicable, but a simpler and, to some extent, a similar method was developed which proved applicable and accurate.

SIZE INFLUENCED BY MOISTURE

The size of prunes is in a measure governed by their moisture content. Prunes which may make a certain grade size if not over-dried will lose several points by remaining in the drier beyond the normal length of time. The grower thereby loses when selling on the count-per-pound basis. There is a further loss in tonnage caused by shrinkage due to over-drying. In analyses made of bin samples collected during the past two years a variation of from 15 to 25 percent in moisture of the flesh was observed. It was also noted that the prunes of the larger sizes uniformly carried the most moisture.

STANDARD MOISTURE FOR STORAGE

Experiments have been conducted by this Station to determine the effect of unfavorable storage conditions on prunes with varying moisture contents. Prunes which have been dried for the purpose of storing until time for packing should not contain in excess of 20 percent moisture for good keeping. Samples were collected at different driers and moisture tests made. These prunes were stored in a room kept at a constant temperature of 86 degrees Fahrenheit and humidities varying from 30 to 75 percent. It was noticed that those samples having moistures below 20 percent kept best with no spoilage, while most of the samples with moisture in excess of 20 percent molded. While molding did not occur in the case of all the prunes having moisture slightly in excess of 20 percent, the percentage of spoilage was sufficiently high to warrant the adoption of 20 percent as a standard.

OFFICIAL MOISTURE-DETERMINATION METHODS

The usual method for the determination of moisture in fruit is too slow for dehydrater and packing-house purposes. The official method for the determination of moisture in dried fruit is to weigh carefully a prepared sample of the fruit in an aluminum or a porcelain dish. The dish is then subjected to a temperature of 100 degrees centigrade in

a controlled oven for from 4 to 5 hours. At the end of this period the dish and its contents are removed to a dessicator to cool, after which it is reweighed and the percentage of moisture computed.

Another method, in which there is less danger of error through loss by driving off the water of crystallization or changing the constitution of the product, is to heat the sample in an oven in vacuum at 60 to 65 degrees centigrade for 24 hours. The sample is then reweighed and the percentage of moisture calculated.

SIMPLIFIED MOISTURE DETERMINATION METHODS FOR PRUNES

Historical. Marcusson[1] was the first to determine moisture by distilling the product which was to be tested with a liquid not miscible with water. A sample of the product was introduced with a quantity of zylene into a flask and distilled into a calibrated receiving tube sealed at the bottom.

Water having a greater specific gravity than xylene settled to the bottom of the measuring tube. There was a distinct line of separation between the water and the xylene. The product was distilled until several hundred cubic centimeters had passed over. Then the column of water was measured on the graduated tube.

Rogers[2] in his work with leather proposed the use of toluene in place of xylene. He used toluene in the same manner as Marcusson had used xylene. Dean and Stark[3] devised a reflux apparatus in which the sample was refluxed with a liquid immiscible with water. The receiver was a calibrated sedimentation tube with a side arm for returning the liquid to the flask. Bidwell and Sterling[4] used a tube which was reduced in diameter so that the column could be read more accurately. These investigators found that their apparatus could be used for the determination of moisture in a number of products where extreme accuracy was not necessary.

The apparatus used by Bidwell and Sterling was the most accurate. It consisted of a 250-c.c. Pyrex Erlyenmeyer flask in which the sample and toluene were placed. This was connected to a reflux sedimentation tube to which a calibrated section from a 5-c.c. Mohr pipette had been attached. A condenser was attached above this tube to condense the toluene and moisture. After filling, the liquid was brought to a boil and distilled at the rate of two drops per second until most of the water had passed over. The rate of distillation was then increased to four drops per second until complete.

The method herein described, although it incorporates some of the essential features worked out by Bidwell and Sterling, is an improvement over that used by these investigators. The Station believes that in introducing this method to the prune growers and packers it has presented a method which will give sufficiently accurate results to meet the needs of these men as well as inspectors and laboratory workers.

Apparatus and chemicals. The apparatus consists of a tube—similar to that described by Bidwell and Sterling—made by using a Pyrex test-tube 1¼-by-6 inches to which is attached a 9-inch graduated tube with glass pet-cock. This is graduated in percent direct. A side arm is con-

nected to the test-tube to allow the condensed toluene to return to the flask.

When using this tube it was found that there is a tendency for the moisture to ride on the surface of the toluene in the tube and return to the flask rather than sink to the bottom of the calibrated tube. To overcome this feature a glass cylinder of smaller diameter than the test-tube is inserted to prevent the droplets of water from returning to the flask (Fig. 1).

A Hopkins condenser is used for condensing the vapors as this condenser keeps the water dropping into the tube, where it collects without attaching itself to the side walls. A 500-c.c. short-neck, round-bottom Pyrex flask is used to boil the sample in. Heat is supplied through a coil of 350 to 400 watts.

A balance type of scale is used having a sensitivity of .1 gram.

A meat grinder for the purpose of grinding the sample into finely divided particles serves for mascerating the sample.

Toluene was used in preference to xylene because of the difference of boiling points. Toluene boils at 114 degrees centigrade and xylene at 139 degrees. The higher temperature drives off water of crystallization, caramelizes the sugars, and causes other chemical changes of the product.

Testing apparatus. Before determining the moisture in prunes a test was first made of the equipment to determine its accuracy of graduation. Tested with distilled water at a constant temperature a variation of .01 percent was found to occur.

Comparison of methods. Moisture determinations made on prune flesh mascerated by passing through the grinder several times and thoroughly mixed gave the following results.

Apr. 18. Italian prunes	Oven test 65° Cent.		Test with Toluene in testing tube		Difference
	(All figures are percents.)	Average		Average	
	13.13	.....	13.15	.....	.....
	13.27	.....	13.00	.....	.....
	.....	13.20	.....	13.075	0.125
	23.74	.....	23.50	.....	.....
	23.35	.....	23.70	.....	.....
	.....	23.55	.....	23.60	0.05
	16.44	.....	16.20	.....	.....
	16.29	.....	16.00	.....	.....
	.....	16.37	.....	16.10	0.27
	27.95	.....	27.25	.....	.....
	27.93	.....	27.90	.....	.....
	.....	27.94	.....	27.575	0.365

These results incorporate tests made without calculating a deduction for correction of 0.01 percent. The average variation of the tests shows a difference of 0.2025 percent, which after the correction is deducted becomes 0.1925 percent. This variation is within experimental error of oven determination at 65 degrees centigrade.

Determinations were made on dried apples with similar results. It is advisable where the product is very wet to run the test for a total of 40 minutes after the toluene starts to boil.

It was particularly noted that in making the determination in prunes a marked change took place in the flesh after boiling for 40 minutes. A charring resulted consisting of darkening of the flesh from a golden

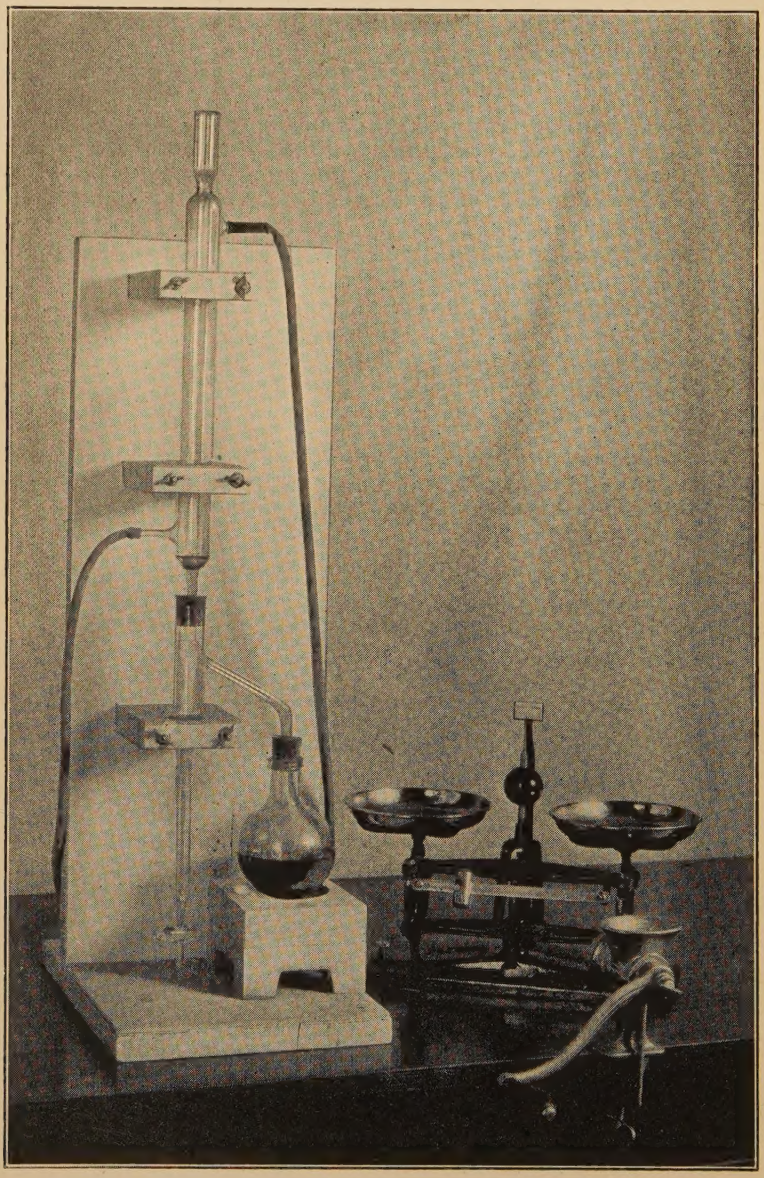

Fig. 3. Assembly of apparatus for moisture determination.

yellow to black. Even boiling for only 40 minutes resulted in a considerable darkening of the tissues. Continued boiling beyond this point caused a change in the product, thus causing an error in the results.

Preparing the sample. When making a moisture test on dried prunes the sample from which the test is to be made should be carefully chosen. If a test is made of a sack of prunes a representative sample of the contents consisting of at least 25 prunes should be taken. These are pitted and the moisture of flesh and pits determined separately. After pitting, run the flesh of the fruit through the grinder at least twice. Thoroughly mix and weigh out 20 grams.

Making the determination. Previous to this the apparatus is carefully set up as illustrated (Fig. 3). The 20-gram sample is then introduced into the 500-c.c. flask and about 200-c.c. toluene is added. The flask is then carefully attached to the delivery tube, precaution being taken that the cork fits tightly. Start the heat by turning on the electric burner and at the same time see that water is circulating freely through the condenser. It takes about five minutes for the toluene to come to a boil. Boil vigorously for thirty minutes. At the expiration of the distillation time turn off the burner and allow a few minutes to cool so that no more toluene will go over. Before making a reading on the tube see that all adhering drops of moisture have settled into the tube.

Making the reading. When making the reading of water in the tube be sure to read the surface of the line of separation between the water in the bottom and the toluene above when the tube is held in a vertical position. The water will adhere to the side of the tube and indicate a higher level unless read in the proper manner. Fig. 2 shows the proper method for reading. The tubes are calibrated to read direct in percent, each division representing one-half of one percent; if read closely moisture can be determined within one-fourth of one percent.

To avoid errors where accuracy is desired duplicate determinations should be made to check on results and the average taken.

Care of apparatus. When making moisture determinations care should be taken to see that the apparatus is clean and dry. This is essential since dirty apparatus will cause considerable variation in the results and error in percent of total moisture in the fruit.

To clean the glassware use good soap suds and Bon Ami. A test-tube brush is used for cleaning the inside of the tube. This tube should be cleaned at least every third test. Between every test the moisture adhering to the walls of the tube can be removed by an applicator made of wire and absorbent cotton. With this it is possible to remove the moisture clinging to the tube, thus avoiding errors in reading.

Removing moisture from toluene. Toluene should be tested for moisture. Any moisture that it contains will cause an error in the determination if it is not removed by distillation. Fill the flask about two-thirds full of toluene and connect up the apparatus as illustrated (Fig 2). Turn on the current and distill for an hour. Moisture which collects in the bottom of the tube can be drawn off through the petcock.

ACKNOWLEDGMENTS

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